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CHLORIDE ANALYSIS IN HAN-BASED LIQUID PROPELLANTS  
BY INDUCTIVELY COUPLED PLASMA SPECTROSCOPY

MADELYN M. DECKER

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<p>Inductively coupled plasma (ICP) spectroscopy was used to determine the chloride content of HAN-based liquid propellants in the parts per million (ppm) range with a detection limit of 1.5 ppm. Chloride is traditionally determined titrimetrically or gravimetrically with a typical precision of 0.01 weight percent (100 ppm).</p> <p>Samples of propellant as formulated and spiked with chloride ions were reacted with a known excess of silver. After the silver chloride precipitate was removed, the remaining silver in solution was analyzed by ICP. This excess metal reaction is necessary in ICP analysis because the instrument is not capable of monitoring emissions from chloride ions. Bromide, iodide, sulfide, cyanide and thiocyanate ions are known</p> <p>SEE REVERSE SIDE FOR CONTINUATION</p>					
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to interfere with this method. Therefore a negative or below detection limit on a chloride analysis would also indicate a negative for the interfering ions since the solubility products of the silver salts of these ions are equal to or below  $1 \times 10^{-10}$  moles per liter.

The method described here is also adaptable for any anion which combines with a metal ion to form a precipitate whose solubility product is less than that mentioned previously. Sulfate determination using an excess of barium ions and carbonate using an excess of lead are two examples. In each analysis, interferences from other anions in the propellant as well as from the propellant itself must be monitored.

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## I. INTRODUCTION

The hydroxylammonium nitrate (HAN) based liquid propellants considered for use in regenerative guns have shown a sensitivity to impurities.<sup>1</sup> Whether these impurities affect propellant stability or its performance in the gun has not been established in all cases. In any case, the absence of impurities in the formulation is a paramount consideration in the production, storage and use of the propellant. The analytical methods used to verify propellant purity should be viable in the lowest levels possible to assure a pure product.

The properties and formulations of the liquid propellants using HAN have been discussed elsewhere.<sup>2</sup> For purposes of this report it is sufficient to know that they are ionic and hence corrosive, thus the possibility for contamination is great. Production of the hydroxylammonium nitrate used in the formulation by electrolytic reduction of nitric acid<sup>3</sup> introduces the possibility of contamination from the impurities present in the acid and the increase of these impurities as the HAN is concentrated by vacuum evaporation at 50°C. Nitric acid impurities include chloride (0.08), PO<sub>4</sub> (0.1), and SO<sub>4</sub> (0.5), ppm respectively as listed on the Baker\* analytical data sheet provided with the nitric acid.

Traditionally, chloride has been determined both gravimetrically and volumetrically by addition and precipitation with silver nitrate. The Fajans, Mohr and Volhard volumetric methods all use silver nitrate as a precipitant. The basic differences in the methods are the end point detection and the pH of the solutions. All three methods are based on the insolubility of the silver chloride precipitate.<sup>4</sup> The gravimetric procedure depends not only on the insolubility of the silver chloride but also on its stability.

Methods exist for the analysis of metals in trace ranges in the liquid propellant. Atomic Absorption (AA) and inductively coupled plasma (ICP) spectroscopy<sup>5,6</sup> are prime examples. To date, methods for anion analysis have been limited to standard volumetric and gravimetric procedures listed previously and lack the high sensitivity necessary for verification of the absence of these substances in the parts per million (ppm) range. Of particular interest is the detection of chloride ions which are known to be present in the nitric acid used to produce the HAN.

With the advent of ICP spectroscopy, detection in the parts per billion range is possible for elements with prominent emission lines from 160 to 800 nm.

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\*J. T. Baker Chemical Company, Phillipsburg, N. J. 08865



This emission range covers all elements except the halogens, hydrogen, helium and the noble gases. The number of detectable elements may be extended to the members of the halogen family by borrowing from the classical titrimetric and gravimetric analyses. Using a known excess of silver nitrate, chloride ions are precipitated, the remaining silver is analyzed by ICP spectroscopy, and the chloride content determined by difference. The method has been used in AA spectroscopy. The limiting factor in the case of AA spectroscopy is the detection limit of the instrument. For ICP spectroscopy, in many cases, it becomes the solubility product ( $K_{sp}$ ) of the precipitate. In this case it is the silver chloride precipitate. The  $K_{sp}$  of silver chloride is listed as  $1.0 \times 10^{-10}$  M/L<sup>2</sup> at room temperature. The concentration of the silver ions in solution contributed by the precipitate, an error in the analysis, is calculated as follows:

$$K_{sp} = [Ag][Cl] = 1.0 \times 10^{-10}$$

$$[Ag] = 1 \times 10^{-5} \text{ M/L}$$

$$\text{WHERE } 10^{-5} \text{ M/L} \times 107.88 \text{ g/M}$$

$$= 1.1 \times 10^{-3} \text{ g/L or 1.1 ppm Ag}$$

Since the atomic weight of chlorine is 32.9% that of silver the resultant error in the chloride ion analysis is 0.36 ppm.

## II. EXPERIMENTAL

Two prominent emission lines for silver exist at 328 and 338 nm. Solutions containing ten parts per million of silver were prepared and the sensitivity and detection limits at each wave-length determined using an ARL 3510 ICP spectrometer.\*\* Scans were completed to assure that there were no interferences at each line.

For the first series of analyses, the method of standard addition was used to assure that matrix interferences did not have a strong influence on the analysis. A standard solution of LP 1846 Lot 50-3 was prepared by diluting 27.1646 grams of propellant to 100 mL. Ten mL aliquots of this solution were delivered to each of four 100 mL volumetric flasks with the same class A volumetric pipet. A blank containing all the additions except the propellant was also analyzed. To each of the volumetric flasks 5 mL of concentrated nitric acid and 5 mL of a standard 1000 ppm silver in solution as silver nitrate were added. A standard chloride solution, of 0.001 M was added in increasing quantities to three of the flasks as indicated in Table 1. The solutions were diluted to 100 mL, agitated to ensure the precipitate

-----  
\*\*Applied Research Laboratories, Valencia, California, 91355

formation and centrifuged at 2800 rpm for 5 minutes. The supernatant liquid was carefully separated from the precipitate and read on the ICP using a standard silver curve for the analysis.

TABLE 1. SOLUTION PREPARATION FOR STANDARD ADDITION ANALYSIS OF CHLORIDE IONS

Description	Blank	LP	A	B	C
LP (g)	None	2.716	2.716	2.716	2.716
Nitric Acid 16 M (mL)	5	5	5	5	5
Silver Nitrate 1000 ppm (mL)	5	5	5	5	5
Cl 0.001 M (mL)	0	0	20	30	50

The data were plotted and a least squares linear regression was determined, allowing a comparison of the theoretical and experimental amounts of silver in the solutions.

A second series of dilutions with higher concentrations of both silver and chloride ions was completed in the same manner as above. These are summarized in Table 2.

TABLE 2. SOLUTION PREPARATION FOR HIGHER CONCENTRATIONS OF CHLORIDE IONS

Description	Blank	LP	1	2	3
LP (g)	None	2.716	2.716	2.716	2.716
Nitric Acid 16 M (mL)	5	5	5	5	5
Silver Nitrate 1000 ppm (mL)	20	20	20	20	20
Cl 0.01 M (mL)	0	0	5	10	15

The entire dilution and analysis procedure was replicated to verify the method at the lower concentrations. Eight 100 mL volumetric flasks, each containing 5.0805 g of propellant were prepared. Five samples of propellant were analyzed with a spike of 10 mL of 0.001 M NaCl to each to determine the precision of the method and the accuracy of the chloride spike determination. Two of the propellant samples were

analyzed with 5 and 20 mL respectively of the chloride added. One sample was retained as the propellant blank and an additional water blank was analyzed.

### III. RESULTS AND DISCUSSION

The line verification using a 10 ppm silver solution showed no interferences when the sample was scanned. A detection limit program showed the 328 nm line to be more sensitive. Results are shown in Table 3.

TABLE 3. ICP LINE SELECTION FOR SILVER EMISSION

Line (nm)	Detection Limit(ppm)	Background Equivalence Conc. (BEC in ppm)
338.289	0.012	0.55
328.068	0.009	0.29

While the detection limits of both lines are within three parts per billion, the point at which the analysis reaches a precision of 1.5% (BEC) is almost doubled for the 338 nm line. The calibration curve is linear from 0.01 to 200 ppm for the 328 nm line and from 0.05 to 1000 ppm for the 338 nm line. Analysis for silver content and the calculated amount of silver remaining in solution are listed in Tables 4 and 5.

TABLE 4. LOW CONCENTRATION ANALYSIS OF UNREACTED SILVER

Description	Blank	LP	A	B	C
LP (g)	None	2.716	2.716	2.716	2.716
Nitric Acid					
16 M (mL)	5	5	5	5	5
Silver Nitrate					
1000 ppm (mL)	5	5	5	5	5
mL 0.001M Cl	0	0	20	30	50
Analyzed Ag (ppm) 328 nm	57.78	57.38	32.58	20.76	1.83
Ag used (ppm)			24.80	36.62	55.55
Equiv Cl (ppm)			8.16	12.05	18.28
Calculated Cl (ppm)			7.09	10.63	17.73
Difference (ppm)			1.07	1.42	0.55

The data from the 338 nm line could not be evaluated because the standard addition plot was not sufficiently linear. This could be attributed to the presence of a small amount of silver chloride precipitate which was not separated from the liquid when the sample was centrifuged. Any silver chloride drawn into the nebulizer of the ICP spectrometer would cause the silver analysis to be high. The use of a microfiltration system during the separation would alleviate this problem and should be considered for future analyses.

TABLE 5. HIGH CONCENTRATION ANALYSIS COMPARING TWO WAVELENGTHS

Description	Blank	LP	1	2	3
LP (g)	None	2.716	2.716	2.716	2.716
Nitric Acid 16 M (mL)	5	5	5	5	5
Silver Nitrate 1000 ppm (mL)	20	20	20	20	20
mL 0.01M Cl	0	0	5	10	15
total Ag (ppm) 328 nm	215.5	212.6	160.8	103.3	43.0
Ag used (ppm)			51.8	109.3	169.6
Equiv. Cl (ppm)			17.0	36.0	55.8
Theoretical Cl (ppm)			17.7	35.4	53.2
Difference (ppm)			-0.7	0.5	2.6
Total Ag (ppm) 338 nm	217.6	216.8	162.6	103.6	43.0
Ag used(ppm)			54.2	113.2	173.8
Equiv Cl (ppm)			17.8	37.2	57.2
Theoretical Cl (ppm)			17.7	35.4	53.2
Difference (ppm)			0.1	1.8	4.0

As can be seen from Table 5 the analysis at the 328 nm line is more accurate when compared to the theoretical amount of chloride ions in the solution.

In the final data set, described in Table 6, five aliquots, each containing 5.0805 g of the propellant and 5 mL of a 1000 ppm silver solution, were spiked with 10 mL of a 0.001 M chloride solution and diluted to 100 mL. In addition, blanks with and without LP, and two samples spiked with 5 and 20 mL 0.001 chloride solution were prepared.

TABLE 6. CHLORIDE ADDITION FOR PRECISION DETERMINATION

<u>Number</u>	<u>Description</u>	<u>328 nm line ppm Ag</u>	<u>338 nm line ppm Ag</u>
1	Blank	52.39	50.67
2	Blank + LP	51.57	50.89
3	LP+ 5 ml Cl	47.80	46.77
4	LP+ 20 mL Cl	29.45	28.72
5	LP+ 10 mL Cl	41.69	41.03
6	LP+ 10 mL Cl	41.28	40.69
7	LP+ 10 mL Cl	41.89	41.30
8	LP+ 10 mL Cl	41.38	41.07
9	LP+ 10 mL Cl	41.14	40.57
10	Average 10 mL	41.48	41.20

The results of the analyses in samples 5 through 9 were averaged and used with samples 3 and 4 to produce a regression analysis. This regression analysis was used to determine the actual amount of chloride ions in the propellant. Additionally, the 10 mL spikes, samples 5 through 9 were used to compute the precision of the method.

The deviation for the five matching samples is 0.7% at the 328 nm line and 1.0% at the 338 nm line. While the standard deviation in samples 5 through 9 is within the limit specified by the manufacturer of the instrument, the repeatability is more reliable at the 328 nm line. Lower concentrations of chloride and silver yield more reproducible results and it is recommended that samples high in chloride contamination be diluted to accommodate the analysis.

Calculations based on the 328 nm data indicate the amount of chloride in the LP-1846 Lot 50-03 is 2.9 ppm. This was computed by standard addition of three increasing aliquots of known chloride concentrations, plotting the data and using a least linear square

regression to compute the x intercept. These three different analyses correspond to values of 3.4, 3.2 and 2.1 ppm. These analyses were completed over a period of several months. Analysis at the 338 nm line was varied and did not produce results that were as precise nor as accurate as those at the 328 nm emission line.

Interferences in the analyses will be the same as those in the traditional volumetric and gravimetric methods. Any anion which forms a silver salt with a  $K_{sp}$  sufficiently low will react with the silver and interfere with the analysis. Examples of these interfering ions and their respective  $K_{sp}$  are listed in Table 7.

TABLE 7. CHLORIDE ANALYSIS INTERFERENCES

Interfering Ion	$K_{sp}$ of Silver Salt
Bromide	$5.35 \times 10^{-13}$
Carbonate	$8.45 \times 10^{-12}$
Chromate	$1.12 \times 10^{-12}$
Cyanide	$5.97 \times 10^{-17}$
Iodide	$8.51 \times 10^{-17}$
Oxalate	$5.40 \times 10^{-12}$
Phosphate	$8.88 \times 10^{-17}$
Sulfide	$1.09 \times 10^{-49}$
Thiocyanate	$1.03 \times 10^{-12}$

The ions listed in Table 7 are not expected to be present in the propellant. However, an analysis which verifies extremely low or below detection limit for chloride would also indicate the absence of the interfering ions, a side benefit of this analysis.

#### IV. CONCLUSION

Analysis for chloride contamination in liquid propellants in the parts per million range is possible by the precipitation of the chloride ions with known excess of silver ions and subsequent analysis by ICP spectroscopy. The 328 nm line was found to be more sensitive and more stable than the 338 nm line. The analysis for chloride ions spiked into solutions showed a deviation of 0.7% at the 328 nm line and 1.0% at the

338 nm line. The recovery of the chloride in the spiked samples was within 1.3% of the theoretical amount. The accuracy and standard deviation are all within the 1.5% listed in the specifications by the manufacturer of the instrument.

All anions which form a precipitate with silver and have a low solubility product will interfere with the analysis. These anions are known to be iodide, bromide, sulfide, cyanide, thiocyanate, chromate and carbonate. While the analysis will not differentiate between these, a low or negative chloride analysis would also indicate low or negative analysis for these anions.

Similar analyses could be completed for sulfate by precipitation with excess barium and for carbonate by precipitation with lead. In each case interferences would be caused by several anions whose solubility products are low. Since ppm is a weight relationship, limits would also be imposed by the weight of the anion being determined.

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